PATENT SPECIFICATION

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Inventors: PETER HUGH VERNON DAWSON, HERBERT ARTHUR DIGGLE, FREDERICK GEORGE HELLYER, HOWARD BEARDALL REVILL and ALAN NEAL.

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COMPLETE SPECIFICATION

NO DRAWINGS

Improvements in or relating to Fibrous Sheet Material

We, THE BRITISH UNITED SHOE MACHINERY COMPANY LIMITED, a British Company registered under the Companies Acts 1862-1898 of Union Works, Belgrave Road in the City of Leicester, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention is concerned with improvements in or relating to fibrous sheet material.

One of the various objects of the present invention is to provide improved fibrous sheet material suitable to provide uppers for shoes and as a substitute for leather in the manufacture of other articles; the word "shoe" is used herein generically to indicate outer footwear generally.

There have been many attempts to provide material other than leather that would be suitable for the uppers of shoes and the uppers of some shoes nowadays are made from materials other than leather. Apart, however, from those shoes which have uppers made of canvas, rubberised woven fabric or decorative textile materials because leather is not as suitable as these materials for the purposes for which the shoes are going to be worn, the vast majority of shoes have leather uppers or uppers made from a material that is recognised as inferior to leather in shoe-making qualities but has been selected because it is cheaper or more readily handled in sheets of uniform size and quality. Among the disadvantages of using leather for shoe uppers is its variable quality and shape when supplied to the shoe manufacturer, and it has long been recognised that a factory-made material of uniform quality comparable with leather both in its suitability for shoe making (e.g. in its ability to take the shape of the last, in its strength and flexibility, in its satisfactory response to skiving and stitching, and in its ability to be bonded by adhesives) and in the satisfaction it can afford the wearers of the shoes by way of comfort and pleasing appearance (which depend on, for example, water vapour permeability, thermal insulating properties, abrasion resistance and suppleness) would be of great value to shoemakers and other users of leather if for no other reasons than would follow from the regular supply of rolls of material of uniform quality, width and thickness.

It is another of the various objects of the present invention to provide a process of manufacturing improved fibrous sheet material of uniform quality and thickness that possesses some of those qualities, e.g. ability to conform to the shape of a last when subjected to the forces imposed on upper material by conventional shoe making machinery, than hitherto have been regarded as characteristic of leather and of few, if any, synthetic materials.

Four sheet materials are hereinafter described in detail to illustrate the invention in its material aspects by way of example, these four illustrative materials having been made by methods that are severally illustrative of the invention in its method aspects. In carrying out these illustrative methods, batts of carded fibres are lightly needled on a needleloom and then assembled in layers and further needled together until a dense thick cross-laid batt of fibres is obtained

in which the fibres are intimately held together by some of the fibres having been forced into and through the batt from both sides, the batt being subsequently impregnated with a bonding agent, dried, and split into thin sheets. The outside splits from the batt may be rejected due to lack of uniformity, but the inside splits, which exhibit a high degree of tear strength and resistance to delamination and in which a proportion of the fibres can be seen to be disposed in bunches perpendicular to the surfaces, are subjected to further processing in the manufacture of shoe upper material.

Fibres used for laying batts in carrying out a method in accordance with the invention are preferably synthetic fibres of high tensile strength selected, for example, from linear polyester fibre (e.g. "Terylene"), nylon, polypropylene, and acrylic staple fibre (e.g. "Acrilan") ("Terylene" and "Acrilan" are registered Trade Marks), or a mixture of these fibres, but the batts may consist of a mixture of such fibres with fibres of natural or synthetic origin and of lower tensile strength, for example viscous, wool, regenerated protein fibre (e.g. "Fibrolene") (Registered Trade Mark) or alginate fibres. A staple length of from 3 ins. to 7 ins. is desirable if the fibres are to be mechanically laid down from a carding machine, but the batt may be air-laid if preferred. Yarn in the form of twisted strands of undefined staple length may also be used. Fibres of 3 denier may be used, but finer fibres of 1½ denier or courser ones (apart from yarn) up to 26 denier may also be used; a blend of fibres of different denier may be selected, bearing in mind that the courser fibres will tend to give high strength to the material while finer ones yield a material having a smoother feel and with the fibres more effectively interlocked after needling, and thus with less tendency to delaminate.

It is desirable, in order to provide adequate water vapour permeability in the finished product if it is intended for shoe uppers, that, unless a bonding agent having water sensitive properties is going to be used, some of the fibres are water sensitive. Water sensitive fibres customarily have low tensile strength and though they may be blended with fibres of high tensile strength in making the batt initially, it may be preferred to introduce water sensitive fibres into the batt by embedding them therein in the form of flock. Whereas in carrying out the illustrative methods, flock is not introduced into the material, material prepared as described in carrying out the first illustrative method, but with flock spread on to both sides of the batt and needled thereinto (one side being treated first and then the other) may, if desired, be made and split down the centre to provide two similar sheets. Alternatively, the material obtained by carrying out the first of the illustrative methods may be split before impregnation and flock embedded into the surface of the splits.

The term "flock" is used herein to denote fibres that are so short as to be free-flowing on handling or in an electrostatic field, being distinguishable from powder in that the ratio of their length to thickness is about as high as it reliably can be for the particles to retain this free-flowing characteristic. A convenient length for flock is of the order of ½ mm for 3 denier filaments, or filaments generally within the range .3 mm for 1½ denier to 1 mm for 8 denier. Viscose, protein and alginate fibres are suitable water sensitive fibres for use in making material in accordance with the invention, and where used in the absence of a water sensitive bonding medium, are preferably present in a proportion by weight of from 30 to 100 parts to 100 parts of non-water sensitive fibres. The presence of flock deeply embedded in one side of the fibrous sheet increases the fibre density and militates against piping of the surface on flexing of the finished material.

Bonding agents suitable for use in carrying out a method in accordance with the invention are flowable compositions based on synthetic rubber (e.g. neoprene or butadiene-acrylonitrile copolymer), a polyurethane or, if water sensitivity of the bonding agent is required, polyvinyl alcohol reacted in situ with sufficient formaldehyde to provide a reaction product insoluble in water but still capable of absorbing and transmitting water vapour, a solution of collagen, or a water sensitive polyurethane, e.g. a reaction product of polypropylene glycol, toluene diisocyanate and polyvinyl alcohol. A convenient range of proportion of bonding agent to fibre in carrying out a method in accordance with the invention is from 20 to 150 parts by weight of bonding agent to 100 parts by weight of total fibre (including any flock present).

Material in accordance with the invention may be treated on one side, which will normally, in the case of material intended for shoe uppers, be the one intended for the outside of the shoe (or "grain" side) and which, in the case of material flocked as hereinbefore described, will be the flocked side, with an additional bonding agent. Such additional bonding agent may be a rubbery polyester, e.g. "Vulcaprene" ("Vulcaprene" is a Registered Trade Mark) or polyurethane, e.g. a reaction product of polypropylene glycol and toluene diisocyanate. The material may also be coated with conventional leather finishes including, for example, an aqueous acrylic resin dis-

persion to provide a wear resistant surface.

Alcohols suitable for forming polyurethane prepolymers for use either as initial bonding agents or for additional surface impregnation in making material in accordance with the invention are preferably dihydric polyethers (e.g. polythylene glycol or polypropylene glycol) having a molecular weight of not less than about 600 and preferably in the range 1000 to a little over 2000 and a preferred diisocyanate is 2-4 toluene-diisocyanate (as available from E.I. Du Pont de Nemours & Co. under the trade name Hylene T) though mixtures, such as Suprasec EN available from Imperial Chemical Industries Ltd. and containing 2-4 and 2-6 toluene diisocyanate, are satisfactory. A small proportion of trihydric alcohol may, however, be included in the reaction mixture to promote limited cross-linking. Preferably, a molar excess of diisocyanate is used so that the prepolymer formed has terminal isocyanate groups. The prepolymer composition thus obtained, where used for surface treatment, may be applied in one coating only, or a second coating may be applied after the first has cured or partially cured. Polyvinyl alcohol may be included in the polyurethane prepolymer composition if desired.

Needles used for needling batts in making material in accordance with the invention are preferably straight and have cylindrical shanks of from 0.015 to 0.036 ins. diameter, the needles being not more than 3½" long and tapering over about the last quarter of an inch to a sharp point. Preferably, also, where the taper joins a cylindrical shank part of the needle, there is a barb provided by a nick from 0.001 to 0.008 inch deep with a kick-up not exceeding 0.001 inch.

Whereas, as described in carrying out the third illustrative method, the needles are straight and supported with their shanks vertical in a needleloom with a vertically reciprocating platen, the needles described may, if desired, in carrying out a method in accordance with the invention, be mounted on the platen with their shanks inclined at an angle of 5 to 10° to the vertical, one set of needles inclined towards another across the width of the loom, so as to reduce the tendency of the material to spread along the direction of feed.

Shoe uppers made from the illustrative materials have been found capable of being conformed to the shape of a last when operated upon by conventional shoe making machines and to share, in common with good quality leather, little evidence of surface delamination such as results on flexing in "piping" or what is commonly called "a bad break." Material which has had flock embedded in one side also exhibits a high resistance to scuffing when lightly abraded on its flocked side. In carrying out the fourth illustrative method, nylon fibres are used, obtained from bulked nylon yarn having a false twist (imparted by twisting nylon filaments, heat setting, and untwisting the filaments) and capable of stretching reversibly to about five times their unstretched length, the yarn having been chopped into six inch staple lengths, opened, carded and needled. A thick densified batt of fibres is obtained which is subsequently impregnated with a neoprene composition, the composition cured, and the batt split into sheets of suitable thickness for shoe uppers. The material obtained has a high ultimate tensile strength and a high degree of resistance to delamination, and also possesses a soft handle and a limited degree of stretchiness. Other bulked yarns, e.g. "Terylene" and polypropylene, may be used.

There now follows a detailed description of the illustrative methods aforementioned and the illustrative materials made thereby. It will be realised that these illustrative methods and illustrative materials have been selected for description by way of example and not of limitation of the invention.

In carrying out the first illustrative method, six batts of cross-laid carded 3 denier 2¼" staple length high crimp Terylene fibres are formed, each batt weighing 500 grams per square yard and consisting of the cross-laid webs of carded fibres. These batts are lightly needled in a needleloom provided with felting needles identified as 15 X 18 X 32, 3" long Regular Barb Spacing with the projecting parts of the barbs filed off, each batt being passed six times through the loom alternately one way up and then the other to effect a density of needle punching of about 1440 needle punches per square inch.

Next, in carrying out the first illustrative method, the batts are impregnated with what will be referred to for convenience as a lubricant, the lubricant being one of the following compositions:—

Lubricant A: 30% solution of mineral oil (machine oil having a viscosity of 230 centipoises at 25°C) in petrol.

When using Lubricant A in carrying out the first illustrative method, the petrol is allowed to evaporate to leave an impregnant deposit of 20-50% on the weight of the unimpregnated batt.

Lubricant B: A solution in water of Manucol SA/LN to provide a viscosity of 230 cps at 25°C. 2% Teepol (on the weight of the solution) is included in the solution. Manucol SA/LN is a solution of sodium alginate.

Where Lubricant B is used in carrying out the first illustrative method, the batt is not dried and the wet impregnant repre-

sents 100-150% of the weight of the unimpregnated batt.

The batts are now, in carrying out the first illustrative method, assembled in two piles, three batts in each, and each pile is passed twice through the needleloom, first one way up then the other, and the two piles are then laid one over the other and the thus assembled batts are passed sixteen to thirty-two times through the loom alternately one way up and then the other. This needling of the thick batt, in the building up of which the carded fibres have been cross-laid, effects a densification of the fibres which is found to be assisted by the lubricant which also appears to reduce fibre breakage. After densification, the needle punch density of the thick batt is from about 5,000 to about 9,000 punches per square inch depending on the number of passes through the loom. Before densification of the thick batt in the needleloom, the weight of the batt was 3.2-3.5 kgs. per square yard; after densification, due to spreading of the batt, the weight is 2.2-2.9 kgm. per square yard and the fibre density 0.34 to 0.43 grams per cubic centimetre. The thickness of the densified batt is 0.25-0.4 inch, and it is observed that a considerable proportion of the fibres are oriented in bunches perpendicular to the surfaces of the batt.

The lubricant is now, in carrying out the first illustrative method, extracted from the thick densified batt by hanging the batt up and allowing petrol or trichlorethylene (in the case of Lubricant A) or water (in the case of Lubricant B) to pour down the material and wash the lubricant away. The batt is then dried. It is also desirable, to safeguard against damage to the splitting machine, to examine the batt at this stage for broken needles by means of X-rays.

The thick batt is next, in carrying out the first illustrative method, impregnated with a bonding agent having the following formulation:

	Parts by Weight
Neoprene latex 750 (50% aq)	200
Vulcastab LW (10% aq)	15
Hylene MP dispersion	27.5
Zinc Oxide (50% aq dispersion)	15
ODN dispersion	50

The Hylene MP dispersion of this composition has the following formulation:—

	Parts by Weight
Hylene MP	100
Casein (12½% aq)	30
Manoxol N (6% aq)	10
Teepol	3
Water	107

and is given at least 48 hours ball-milling before being used. This Hylene MP dispersion has a solids content of 43% (Hylene MP 40%). Hylene MP is a blocked diisocyanate, viz. a bis phenol adduct of methylene bis (4-phenylisocyanate), obtainable from E.I. Du Pont de Nemour & Co., Inc. "Hylene" is a Registered Trade Mark.

The ODN dispersion of this composition is made up by pouring 50 parts by weight of ODN with 2.5 parts by weight of oleic acid slowly into 45 parts by weight of water with 2.5 parts by weight of 0.88 ammonia, stirring rapidly. "ODN" is an adipate type plasticizer obtainable from British Industrial Solvents Ltd.

The composition is 47.4% solids and is thickened with Manucol SA/LN. Manucol SA/LN is a sodium alginate solution, and the desired viscosity of the composition in carrying out the first illustrative method, measured at 20°C on the Brookfield viscometer, is adjusted to 2.500 centipoises at speed 2 with spindle 6 and 2,200 centipoises at speed 20 with spindle 6. Vulcastab LW is a stabilizer for the neoprene latex. The batt is dried at 50°C and the neoprene is cured by heating the batt in an oven at 120°C for 45 minutes. The weight of dried impregnant is approximately equal to that of the fibre.

The cured batt, next in carrying out the first illustrative method, is split on a band knife splitting machine. The outside layers will probably be rejected as not sufficiently uniform in thickness and quality, and it is found convenient to split them off at about 0.020" thickness. The inner layers are split at thicknesses selected for the purpose for which the material is to be used. For upper material for men's wear splits of 0.070-0.080 inch are suitable; for women's wear 0.035-0.040 inch. The tear strength of the splits is found to be of the order of 12 to 23 lbs per inch width per 100 grams per square yard of the material and the material is found to have a good "break" and to exhibit a high degree of resistance to delamination.

Where this illustrative material is to be used for shoe uppers, it is now subjected to further suitable treatment; for example the material may be buffed and coated on what will be regarded as the "grain" side with a surface-treating composition containing a rubbery polyester type material such as Vulcaprene AC 50 and provided with a conventional leather surface finish.

It will be appreciated that if desired the thick densified batt obtained in carrying out the first illustrative method may be split before impregnation with the bonding agent and the individual splits then treated by impregnation with a bonding agent after, if so desired, rubbing flock into one surface. Suitable flock is 3 denier ½ m.m. cut viscose rayon flock, and it may be spread

over the surface of the splits and rubbed into the surface by hand. The distribution of the flock may be 150 grams per square yard.

In carrying out the second illustrative method a split sheet of weight 600 grams per square yard and thickness 0.060 inch obtained by impregnating with neoprene a densified needled batt of Terylene fibres and splitting the sheet in the manner followed in carrying out the first illustrative method is coated on one side with a prepolymer composition obtained as follows:—

	Parts by Weight
Niax Diol PPG 1025	2350
Toluene diisocyanate	800

Niax Diol PPG1025 is a polypropylene glycol of molecular weight 1025 obtainable from Union Carbide Ltd. The glycol and toluene diisocyanate are mixed and allowed to react for five hours at 90°C in an atmosphere of nitrogen. The proportions chosen are such that the ratio of isocyanate groups to hydroxyl groups is 2.1. A polyurethane prepolymer of flowable consistency is obtained; a suitably coloured filler may be added. The composition is spread on one side of the split sheet by means of a doctor blade to give a uniform coating of 100 grams per square yard. The coating is allowed to cure in air at room temperature; if desired .15% by weight of catalyst, such as dibutyl tin dilaurate, based on the weight of prepolymer may be included in the coating composition, and the sheet warmed to speed up curing of the prepolymer.

After the prepolymer composition has cured, in carrying out the second illustrative method, the sheet is buffed on the coated side and then further coated by application of a composition having the formula:—

	Parts by Weight
Vulcaprene AC160	5
ECR Clay	5
Acetone	95

Vulcaprene AC160 is a rubber polyester obtainable from Imperial Chemical Industries Ltd.; ECR clay is obtainable from English China Clay Co. Ltd. "Vulcaprene" is a Registered Trade Mark. The composition may also include Desmodur R, a curing agent for the Vulcaprene.

This composition is allowed to dry and an acrylic resin dispersion of the following formulation is then applied to the coated side of the sheet:—

	Parts by Weight
Primal B41	50
Insoluble Starch grains (G7443)	5
ECL Clay	4
Ben-a-gel dispersion	50
Water	95

Primal B41 is a 40% aqueous acrylic resin dispersion obtainable from Rohm and Haas. Ben-a-gel dispersion is a 3% aqueous dispersion obtainable from F.W. Berk & Co. Ltd. and understood to contain highly purified magnesium montmorillinite; it serves the purpose of rendering the composition thixotropic. The starch grains have an average grain size of 20 microns.

The sheet is allowed to dry and is pressed with a hot plate at 55°C and is thereafter coated, in further carrying out the second illustrative method, with three applications of a dispersion of acrylic resin of medium hardness and a final finish of nitrocellulose, the coatings being allowed to dry separately and the sheet being plated after the first and last of the acrylic resin applications.

The illustrative material which results from carrying out the second illustrative method has a high degree of tensile strength and resistance to delamination and has a pleasing appearance and acceptable scratch resistance. A piece of this illustrative material cut to the shape of a shoe upper and used for making a shoe on conventional shoe making machinery is capable of conforming to the shape of the shoe last.

The third illustrative method is carried out in the same manner as the first illustrative method except that instead of providing the needleloom with the felting needles there identified, the needles are characterised by having a round smoothly polished nickel-plated shank 2.6 ins. long and .024 ins. diameter terminating in a sharp point to which the shank tapers along a gradual curve a quarter of an inch long, a barb about a quarter of an inch from the point being provided by a nick .005 in. deep with a kick-up projecting not more than .001 ins. from the needle shank; the needle has only the one barb. Conveniently, the needles are obtained by nicking domestic sewing needles of the given dimensions. The needles are mounted point downwards in the needleloom with their upper ends buried in a polythene block; they may be more closely spaced than on a conventional needleloom. After penetrating the batt in the operation of the needleloom loom, the points of the needles are received in a felt underlay.

Needling of the thick batt in carrying out the third illustrative method is proceeded with until the density of punching is of the order of 20,000 to 40,000 punches per square inch. The thickness of the resulting densified batt is 0.3 to 0.5 inch. The batt is next impregnated, in carrying out the third illustrative method in the manner followed in carrying out the first illustrative method using the neoprene composition there identified or a similar composition but with additional filler, for ex-

ample 200 parts by weight of china clay introduced as a slurry with 30 parts of a 1% aqueous solution of Vulcastab LW to the 200 parts of neoprene latex. Other suitable fillers may be used instead including water sensitive ones, e.g. finely divided cellulose. The impregnant is cured and the batt thereafter is split into sheets which are buffed coated and finished on one side in the manner followed in carrying out the second illustrative method.

The resulting material is found to have a fine structure in which some fibres are disposed depthwise through the material but not in large bundles, and the material has a high degree of tensile strength and resistance to delamination. A piece of this illustrative material cut to the shape of a shoe upper and used for making a shoe on conventional shoe making machinery is capable of conforming to the shape of the shoe last.

Whereas in carrying out the third illustrative method, the thick needled batt is split, after curing of the impregnant, into a number of sheets, as an alternative a batt of fibres may be needled with either the felting needles used in carrying out the first illustrative method to a density of about 9,000 punches per square inch, or with the finer needles just described, and to a thickness of 0.15 to 0.25 inch, buffed and impregnated; the material may then be cured in a press at 120°C for 35 minutes under a pressure of 300 lbs per square inch to reduce the batt to a thickness of 0.08 to 0.12 in such that its density is 16 gms. per square yard per .001" of thickness. Such batt may then be split through the middle and the split face buffed, coated and finished in the manner followed in carrying out the second illustrative method. Or, alternatively, if a material of thickness greater than half the thickness of the batt is required, the batt may be split to provide one sheet of material of such thickness as is required.

In carrying out the fourth illustrative method, the procedure adopted in carrying out the third illustrative method is followed, but instead of using 3 denier 2½ inch staple length high crimp Terylene fibres, highly stretchable nylon fibres 2 denier with a false twist and 6 inch staple length are carded, cross laid, lightly needled and assembled for densification as a thick batt by needling with the fine needles. Alternatively, the thicker multi-barbed needles of the first and second illustrative methods may be used. The densified batt is impregnated, the impregnant cured, the batt split, and the resulting sheets buffed, coated and finished as in carrying out the second illustrative method.

The resulting material, which is also illustrative of the invention in certain of the material aspects, has a high ultimate tensile strength and high degree of resistance to delamination; it has a soft handle and a limited degree of stretch. A piece thereof cut to the shape of a shoe upper conforms to the shape of a last when a shoe incorporating such piece is made in conventional shoe machinery.

WHAT WE CLAIM IS:—

1. A method of preparing a non-woven web of fibres having relatively high tear strength and resistance to delamination comprising the steps of (a) needling a batt of fibres to cause them to become intimately intermingled, (b) bonding the fibres together by impregnation with a bonding medium, and (c) splitting the batt to provide at least two separate sheets.

2. A method according to Claim 1 wherein the batt is split to provide a plurality of sheets in addition to two separate exterior sheets split one from each side.

3. A method according to either one of the preceding claims wherein the batt of fibres, before needling, if formed by cross-laying a carded web of the fibres.

4. A method according to any one of the preceding claims wherein the batt comprises synthetic fibres of high tensile strength.

5. A method according to Claim 4 wherein the batt comprises linear polyester fibres.

6. A method according to any one of the preceding claims wherein the bat comprises fibres of 1½ to 5 inch staple length.

7. A method according to Claim 4 wherein the batt also comprises fibres of natural or synthetic origin and of low tensile strength.

8. A method according to any one of the preceding claims wherein some of the fibres of the batt are water sensitive and some are non-water sensitive.

9. A method according to Claim 8 wherein the proportion of water sensitive fibres in the batt is 30-100 parts by weight to 100 parts by weight of non-water sensitive fibres.

10. A method according to any one of the preceding claims wherein the needles used have cylindrical sharply pointed shanks of from 0.015 to 0.036 inch diameter.

11. A method according to Claim 10 wherein the needles used have a barb provided by a nick from 0.001 to 0.008 inch deep with a kick-up not exceeding 0.001 inch.

12. A method according to any one of the preceding claims wherein the batt is impregnated with a lubricant that assists a subsequent needling operation.

13. A method according to any one of the preceding claims wherein the bonding agent used is a polyurethane.

14. A method according to Claim 15 wherein the bonding agent is a reaction pro-

duct of polypropylene glycol and toluene diisocyanate.

15. A method according to any one of the preceding claims wherein the batt of fibres comprises fibres obtained from bulked yarn.

16. A method according to Claim 15 wherein the yarn is one having a false twist imparted to it.

17. A method according to any one of the preceding claims wherein to one side of a sheet split from the batt is applied a flowable coating composition comprising polyurethane prepolymer, the composition being allowed to solidify and the prepolymer to cure.

18. A method according to Claim 17 wherein the prepolymer is a reaction product of polypropylene glycol and toluene diisocyanate.

19. A method according to any one of the preceding claims wherein the batt is impregnated with a solution of collagen which is caused to precipitate among the fibres of the batt.

20. A method of preparing a non-woven web of fibres having relatively high tear strength and resistance to delamination comprising the steps of (a) needling a thick batt of fibres to cause them to become intimately intermingled, (b) splitting the batt to provide at least two separate sheets and (c) impregnating the separate sheets with a bonding agent.

21. A method according to Claim 20 wherein, after splitting the batt, flock is applied to the separate sheets and caused to become embedded therein before impregnating the sheets with bonding agent.

22. A method of making sheet material carried out substantially as hereinbefore described as the first illustrative method.

23. A method of making sheet material carried out substantially as hereinbefore described as the second illustrative method.

24. A method of making sheet material carried out substantially as hereinbefore described as the third illustrative method.

25. A method of making sheet material carried out substantially as hereinbefore described as the fourth illustrative method.

26. Sheet material made by carrying out a method according to any one of the preceding claims.

J. W. RANDALL,
Chartered Patent Agent,
160 Belgrave Road,
Leicester.

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